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MEMORANDUM

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FROM: Fred Seto, Ph.D.
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DATE: April 21, 2004

SUBJECT: Boeing Rocketdyne - Santa Susana Field Laboratory RFI
Review of Lockheed Martin Report on Audit of Data Packages
Centrum/Calscience: Methods 8270 -Semi-Volatile Organics (SVOC), 8270 SIM - Polyaromatic Hydrocarbons (PAHs), 8082 - Polychlorinated Biphenyls (PCBs)
Ceimic: Methods 8270 (SVOC), 8270 SIM (PAHs), 8082 (PCBs)

The Department of Toxic Substances Control (DTSC) has contracted with Lockheed Martin (LM) to perform electronic and hard copy audits of the laboratory data. LM audited the electronic and hard copy data for the following data packages:

SDG (Sample Delivery Group)	No. of Samples - Matrix	Method	Laboratory
0028	1 - Soil	8270 (SVOC)	Centrum
0012	13 - Soil, 1 Water	8270 SIM (PAHs)	Calscience/Centrum
0027	2 - Soil	8082 (PCBs)	Centrum
RJ579	4- Soil	8270 (SVOC)	Ceimic
RJ028	9- Soil, 1 Water	8270 SIM (PAHs)	Ceimic
RJ772	16 - Soil	8270 SIM (PAHs)	Ceimic
RJ214	5 - Soil, 1- Water	8082 (PCBs)	Ceimic

The Hazardous Materials Laboratory (HML) of DTSC received and reviewed a LM

report. We have the following summary/comments:

LM performed the audit of the hard and electronic copy with the following approach by using Chemstation or Target software as appropriate:

a. The hard copy was compared to standard EPA Contract Laboratory Program data package requirements.

b. The electronic copy was reviewed to verify that information provided in the hard copy matched the corresponding information in the electronic form.

c. Where the required information was available for GC/MS analyses, the initial calibration for six to ten target compounds, one surrogate and the internal standards were transcribed into a spreadsheet. These values were used to regenerate and verify results for selected quality control and client samples as reported in the hard copy.

d. Chromatograms and GC/MS spectra were examined to verify appropriateness and accuracy of any manually integrated instrument responses.

LM evaluated, as appropriate, the results of the tuning, initial calibration (ICAL), continuing calibration verification (CCV), method blanks, surrogates, internal standards, laboratory control samples/laboratory control samples duplicates (LCS/LCSD), matrix spike/matrix spike duplicate (MS/MSD), retention times (RT) and manual integration of some quality control (QC) samples and client samples and verified some QC and client sample results. The audit results or comments are summarized in Tables 1 to 7.

SDG 0028 - Method 8270 (SVOC) by Centrum

As detailed in Table 1, for SDG 0028, the tuning did not meet the method requirement. The initial calibration (ICAL) electronic and hard copies were not provided. The CCV's hard copy provided in the data package did not match the electronic data (12/21/00).

The method blank (MB), LCS/LCSD, MS/MSD and surrogates appear to meet the quality control limits.

The sample was analyzed with a 1:10 dilution. There was no data for an undiluted sample analysis. There was no indication of any sample extract cleanup. It is not clear why the sample was analyzed with a 10 fold dilution.

The values for the hard copy quality control samples and the actual sample RZ855 were manually integrated. No explanation was provided for using manual integration.

SDG 0012 - Method 8270 SIM (PAHs) by Calscience/Centrum

As indicated in Table 2, for SDG 0012, the tuning, ICAL and CCV hard copy/electronic

data were not provided.

MB hard copy/electronic data and blank summary forms were not provided. It appeared that one blank (water matrix) was used for both water and soil samples. Each matrix should have a MB. The analytical report and quality assurance (QA) summary sheet showed that the MB contained no PAHs above the reporting limits and surrogate recoveries met the QC limits. But without the associated raw data, the auditor cannot confirm the results.

LCS/LCSD hard copy/electronic data were not provided. But the QA summary sheet showed the LCS/LCSD met the QC limits. But without the associated raw data, the auditor cannot confirm the results.

MS/MSD hard copy/electronic data were not provided. The MS data submitted on the CD was not applicable to this batch.

The hard copy for samples RX001-RX014 were not provided. Except for RX014, the electronic *Eptem* files were provided, so the auditor was able to reproduce the quantitation reports and chromatograms for review. But due to missing sample preparation, ICAL, CCV and QC information, the auditor cannot validate any of the sample results.

Electronic data of samples of RX003 and RX004 showed incorrect manual integrations for some phthalates. The auditor was able to use the correct retention times (RT) to identify the presence of some phthalates. However, the concentrations of the phthalates present in the samples were not significant even after integration of the correct peak. On the final report only PAHs were listed as the report format did not include phthalates. The rationale for the manual integrations was not clear. Phthalates are common laboratory contaminants. Since the raw data for method blanks were not provided, source of contamination could not be determined.

Report forms of the internal standard area counts and retention time summaries were not provided.

Sample results were reported in mg/Kg, rather than ug/Kg. The report did not indicate whether concentrations were in wet or dry weight basis. WX014 and MB results were reported in mg/Kg. Since their matrix is water, the reporting units should be ug/L.

<u>SDG 0027 - Method 8082 (PCBs) by Centrum</u>

As shown in Table 3, for SDG 0027, the ICAL hard copy/electronic data and summary form were not provided.

The CCV hard copy was provided but incomplete. It has a quantitation and evaluation report but no chromatograms. The CCVs bracketing the samples were not run within 12 hours of one another as required. This invalidates the sample results.

The hard copy quantitation reports indicate that manual integrations were performed on almost all surrogates and target compounds in the CCV with the primary and confirmation columns. No explanation was provided for using the manual integrations.

It appeared that two MBs were analyzed. The hard copy for both MBs and the electronic copy for the second MB were not provided. It is not clear whether a MB was reported for each sample or only one MB was reported for both samples. The extraction log indicated that the samples, MB, and LCS/LCSD underwent florisil cleanup except samples RZ781 and RZ776. Thus, for samples RZ781 and RZ776, the MB was not prepared exactly as the samples.

LCS/LCSD recoveries and RPDs were within the QC limits. But it was not clear why a clean matrix of LCS/LCSD would use a florisil cleanup.

MS/MSD hard copy/electronic data and the summary data were not provided. Extraction log indicated "not enough sample for MS/MSD." Another sample with " s imilar" matrix could have been used for MS/MSD analysis.

For sample RZ781, only 0.8 g was extracted instead of 30 g that was used for sample RZ776. No explanation was provided for the reduced weight. Detection limit was appropriately raised to reflect the reduced weight.

Aroclors were reported as Non-Detect (ND). The surrogate, tetrachloro-m-xylene, recoveries met the QC limits. No recoveries were reported for the surrogate, decachlorobiphenyl (DCB), although it was spiked and measured (manually integrated) in the client samples and QC samples. DCB did not meet the 15% D requirement on the second CCV.

SDG RJ579 - Method 8270 (SVOC) by Ceimic

As shown in Table 4, for SDG RJ579, the tuning was satisfactory even though the laboratory used tuning criteria specified in the Contract Laboratory Program (CLP) instead of the criteria specified in the method 8270.

ICAL standards manual integrations were appropriate and accurately performed on some standards. The ICAL results were within the control limit of 15% RSD except for four compounds. They are: Pyridine, Benzidine, Benzo(b)fluoranthene, and Benzo(k)fluoranthene. As the method specifies, for RSD >15%, average response factor (RF) cannot be used for quantitation. Calibration curves should have been constructed for the 4 compounds rather than using the average RF. Consequently the results reported for these compounds should be considered estimates because calibration curves were not constructed as required by the method.

The CCV results were satisfactory.

The MB relating to sample RJ587 (reanalysis) showed the presence of Di-n-Butylphthalate (48 ug/Kg) and Bis(2-ethylhexyl)phthalate (42 ug/Kg). The MB relating to

samples RJ592, RJ596, RJ599 showed the presence of Di-n-Butylphthalate (37 ug/Kg). It appeared that these phthalates were laboratory contaminants.

The LCS was satisfactory but no LCSD data was available.

The MS/MSD were not provided. A pair of MS/MSD were extracted and analyzed by method 8270 SIM but results were not submitted with method 8270B.

For sample RJ587, Benzo(b)fluoranthene and Benzo(k)fluoranthene were identified and quantitated. Since the RSD of these compounds exceeded the 15% limit and the average RF was used for quantitation, the reported results should be considered as estimates.

Samples RJ592 and RJ596 contained no target compounds except two low level phthalates. Sample RJ599 contained two low level phthalates and Bis (2-ethylhexyl) phthalate with a concentration of 350 ug/Kg.

SDG RJ028 - Method 8270 SIM (PAHs) by Ceimic

As shown in Table 5, for SDG RJ028, the tuning was satisfactory even though the laboratory used tuning criteria specified in the Contract Laboratory Program (CLP) instead of the criteria specified in the method 8270.

The ICAL results were within the control limit of 15% RSD except for a group of 12 compounds as listed in Table 5. They are: N-Nitrosodimethylamine, Fluorence, Acenaphthylene, Pyrene, Anthracene, Benzo(b)fluoranthene, Benzo(a)anthracene, Indeno(1,2,3-cd)pyrene, Benzo(k)fluoranthene, Benzo(g,h,i)perylene, Dibenzo(a,h)anthracene, and 2-Methylnaphthalene.

The CCV and MB were satisfactory.

The LCS/LCSD recoveries for the water matrix were within the control limits of 20% to 140%. For soil, the LCS recoveries of 4 compounds (N-Nitrosodimethylamine, Anthracene, Benzo(a)pyrene and Benzo(g,h,i)perylene) were below 20%. The LCS recoveries of other compounds were within the control limits.

No MS/MSD data was available to indicate any extraction or analysis.

For the samples, the surrogate recoveries were within the control limits except for sample RJ036 where the recoveries were diluted out due to the presence of high levels of PAHs. Results of target compounds with ICAL >15% RSD should be considered as estimates.

The quality control limits for two surrogates (2-Fluorobiphenyl and Terpheny-d14) and the upper control limit for the internal standard used by the laboratory are not as stringent as the limits required by method 8270. However, the quality control results met the requirements of method 8270.

SDG RJ772 - Method 8270 SIM (PAHs) by Ceimic

As shown in Table 6, for SDG RJ772, the tuning was satisfactory even though the laboratory used tuning criteria specified in the Contract Laboratory Program (CLP) instead of the criteria specified in the method 8270.

The ICAL results were within the control limit of 15% RSD except for 6 compounds as listed in Table 6. They are: Acenaphthylene, Indeno(1,2,3-cd)perylene, Benzo(k)fluoranthene, Benzo(g,h,i)perylene, Dibenzo(a,h)anthracene, and Anthracene.

The CCV and MB were satisfactory.

Three LCSs were analyzed. Two of them had acceptable recoveries for all compounds. One of them had acceptable recoveries for all compounds except for N-Nitrosodimethylamine with recovery value of 18% (just below the 20% lower limit).

The MS/MSD had recoveries and RPDs meeting the control limits, although the N-Nitrosodimethylamine, Dibenzo(a,h)anthracene and Benzo(g,h,i)perylene were at the lower limit (20% R) in the matrix spiked sample.

For samples RJ788, RJ789, RJ790 and RJ791, the surrogate recoveries were diluted out due to the presence of high concentration PAHs. The surrogate recoveries for other samples were satisfactory.

Six samples were analyzed at 5 fold or greater dilution. No reports or raw data were submitted for undiluted extracts.

SDG RJ214 - Method 8082 (PCBs) by Ceimic

As shown in Table 7, for SDG RJ214, the results for the ICAL, CCV, MB, surrogates, LCS/LCSD and MS/MSD were all satisfactory. The retention times were within the RT windows and the software integrations were appropriate. Reported results were satisfactory. The analytical work performed appears to be of very high quality.

Conclusions/Recommendations

For reasons discussed above with the details provided in Tables 1 to 7, HML has reached the following conclusions/recommendations:

1. The results for SDG 0028-Method 8270 (SVOC) by Centrum are not usable.

2. The results for SDG 0012-Method 8270 SIM (PAHs) by Calscience/Centrum and SDG 0027-Method 8082 (PCBs) by Centrum can not be meaningfully evaluated due to incomplete/missing hard copy and electronic data.

3. The results for SDG RJ579-Method 8270 (SVOC), SDG RJ028-Method 8270

SIM (PAHs) and SDG RJ772-Method 8270 SIM (PAHs) by Ceimic are acceptable. However, some compounds, if reported for any sample, should be qualified as estimates due to their ICAL RSD exceeding the 15% requirement. They are as follows:

SDGRJ579: Pyridine, Benzidine, Benzo(b)fluoranthene, and Benzo(k)fluoranthene.

SDG RJ028: N-Nitrosodimethylamine, Fluorence, Acenaphthylene, Pyrene, Anthracene, Benzo(b)fluoranthene, Benzo(a)anthracene, Indeno(1,2,3-cd)pyrene, Benzo(k)fluoranthene, Benzo(g,h,i)perylene, Dibenzo(a,h)anthracene, and 2-Methylnaphthalene.

SDG RJ772: Acenaphthylene, Indeno(1,2,3-cd)perylene, Benzo(k)fluoranthene, Benzo(g,h,i)perylene, Dibenzo(a,h)anthracene, and Anthracene.

4. The results for SDG RJ214-Method 8082 (PCBs) by Ceimic are acceptable. This data package appears to be of very high quality.

If you have any questions, please feel free to contact Lorna Garcia or Fred Seto at (510) 540-3003.

CC: Cindy Dingman
Lorna Garcia
James Cheng
Bart Simmons, Ph.D., Chief

TABLE 1: SDG 0028 AUDIT SUMMARY

QA/QC	SDG 0028 - Centrum - 8270 (SVOC) Analyzed 12/21-22/00 ID No: RZ855 1 Soil
Tuning	First tune (12/21/00, 0824 hrs), did not meet method criterion that Mass 442 must be >40% of Mass 198. The lab criterion was 30% to 100%. Case narrative did not explain alternative tuning criterion. This failure invalidated the data for CCV, MB, LCS/UCSD, and MS/MSD. Second tune (12/21/00, 2018 hrs) was not submitted in hardcopy and the electronic file does not contain a tune evaluation file. The auditor could not verify that the tune met the method acceptance criteria. This tune is applicable to the second CCV, second MB, a LCSD, and the sample RZ855.
Initial Calibration (ICAL)	Hard copy/electronic data - Not provided
Continuing Calibration Verification (CCV)	Not acceptable -hard copy (apparently printed 1/19/2001 after all the compounds had been manually integrated) did not match the electronic file data for first CCV(12/21/00, 0912 hrs). Second CCV (12/21/00, 2106 hrs) applicable to sample RZ855. Hardcopy data generated on 1/19/2001 after all compounds had been manually integrated. Calculation of compounds RFs and %Ds for this CCV were not performed due to overall problems with the data package.
Method Blank (MB)	First MB , a hardcopy quantitation report, generated on 1/19/2001 after all compounds had been manually integrated, met laboratory's QC limits. Second MB, no hardcopy was provided. The quantitation report printed out from electronic file indicated acceptable recoveries for surrogates and the absence of contaminating compounds.
Laboratory Control Sample/Duplicate (LCS/LCSD)	A LCS analyzed first 12 hrs of analytical sequence (12/21/2000, 1000 hrs) prior to client sample. A LCSD was analyzed after the client sample. However, the LCSD was analyzed more than 12 hours after the last instrument tune and CCV standard. Otherwise, the LCS/LCSD met the laboratory's QC limits.
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	All compounds on the hardcopy quantitation reports for MS/MSD had been manually integrated. The indicated results met the laboratory's acceptance limits.
Chain-of-Custody (COC) Forms, Sample Receipt, Extraction, Standard and Instrument Logs	Standard and Sample receipt logs - Not provided
Client Sample:	All internal standard and surrogate spike values for sample RZ855 (17633-1) had been manually integrated on the 2/21/2001 hardcopy quantitation report. The concentrations were reported as ug/L rather than ug/Kg and all target compounds were reported as NDs. No explanation was given for the manual integrations and for the sample being analyzed in a diluted state (1 to 10 dilution) when no target compounds were detected.
Overview Comments	No indication that the sample extract went through any cleanup procedure. The sample was analyzed in an extract with 1 to 10 dilution. There was no data for an undiluted sample analysis. Surrogate recoveries met the QC limits.

QA/QC	SDG 0028 - Centrum - 8270 (SVOC) Analyzed 12/21-22/00 ID No: RZ855 1 Soil
	Internal standard summary sheets were not provided.

TABLE 2: SDG 0012 AUDIT SUMMARY

QA/QC	SDG 0012 - Calscience/ Centrum - 8270 SIM (PAHs) Analyzed: 12/28-30/99 ID. Nos: RX001- RX013, RX014 3 soil, 1 water
Tuning	Hard copy/electronic data , report forms for DFTPP tune - Not provided.
Initial Calibration (ICAL)	Hard copy/electronic data, ICAL summary forms - Not provided
Continuing Calibration Verification (CCV)	Hard copy/electronic data, CCV check forms - Not provided
Method Blank (MB)	Hard copy /electronic data, MB summary forms - Not provided. Analytical report showed one MB was extracted (12/22/99) for water and soil samples. The MB was analyzed (12/28/99) with the water sample. So, it appears no MB for soil samples. Each matrix should have a MB. Analytical report and QA summary sheet showed MB contained no PAHs above reporting limit and surrogate recoveries met the QC limits. Without the raw data, the auditor cannot confirm the results.
Laboratory Control Sample/Duplicate (LCS/LCSD)	Hard copy/electronic data - Not provided QA summary sheet indicated that the LCS/LCSD results met QC limits. Without the raw data, the auditor cannot confirm the results.
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	Hard copy/electronic data, summary data - Not provided The MS data submitted on the CD was not applicable to this batch.
Chain of Custody (COC), Sample Receipt, Standard, Extraction and Instrument Logs	Extraction logs, standard logs, instrument logs - Not provided
Client Sample	No indication of the type of extraction used. Incorrect manual integration of phthalate esters were identified in samples RX003 and RX004. The quantitation reports showed that RT were off by 0.2 - 0.3 minutes while the corresponding ISTD were stable. The electronic data showed that the spectrum of the peak selected and integrated by the analyst did not match the library (reference) spectrum for that compound. The phthalate esters were di-n-butyl phthalate, butyl benzyl phthalate, bis(2-ethylhexyl) phthalate for sample RX003 and butyl benzyl phthalate, bis(2-ethylhexyl) phthalate for sample RX004. The auditor reintegrated the phthalate esters by selecting the correct peak within the established RT and match the spectrum of the selected peak with library (reference) spectrum. The quantity of phthalate esters in the samples were not significant even after integration of the correct peak. The Analytical Report format only listed PAHs, so the rationale for the incorrect manual integration was unclear to the auditor. Phthalate esters are common laboratory contaminants. Without MB's raw data, the source of phthalates whether from soil matrix, solvents and equipments cannot be determined. The 5 phthalate esters were also present in sample RX004 at

QA/QC	SDG 0012 - Calscience/ Centrum - 8270 SIM (PAHs) Analyzed: 12/28-30/99 ID. Nos: RX001- RX013, RX014 3 soil, 1 water
	concentrations from 0.004 to 0.22 mg/Kg. None of the peaks were manually integrated and were not reported evidently because the Analytical Report format only listed PAHs. All other manual integrations were examined and appeared to be correctly performed.
Overview Comments	Hard copy for samples RX001 - RX0014 were not provided. Except for RX014, electronic *Epatem* files were provided, the auditor was able to reproduce the quantitation reports and chromatograms for review. But the auditor cannot validate any of the sample results because extraction log, ICAL, CCV and QC information were not provided. No *Method* file included in the electronic data, so the auditor could not review the method specifications or recreate the quantitative approach. Report forms of the internal standard (IS) area and retention time summaries - Not provided. Sample results were reported in mg/Kg, rather than ug/Kg. No notification on the report whether concentrations were in wet or dry basis. RX014 and MB results were reported in mg/Kg when both are liquid matrices. The reporting units should be ug/L.

TABLE 3: SDG 0027 AUDIT SUMMARY

QA/QC	SDG 0027 - Centrum - 8280 (PCBs) Analyzed 11/19 - 20/00 ID. Nos: RZ776, RZ781 2 - Soil
Initial Calibration (ICAL)	Hard copy/electronic data, ICAL summary forms - not provided
Continuing Calibration Verification (CCV)	Hard copy quantitation report and 3 CCVs evaluation report were provided but no chromatograms. Electronic copy provided has no *Epatemp* files, so auditor cannot reproduce chromatograms The first two CCVs bracketing the samples were not run within 12 hours of one another as required. First CCV run on 11/19/00 at 1324 hrs, sample RZ781on 11/19/00 at 2026 hrs, sample RZ776 on 11/20/00 at 0740 (over 18 hrs) hrs and the second CCV on 11/20/00 at 0947 hrs (over 20 hrs). This invalidates the sample results. Hard copy quantitation reports indicated that manual integrations were performed on almost all surrogates and target compounds in each of the CCV standards with the primary and confirmation columns. *Epatemp*/*method* files of the electronic data were not provided, so the auditor cannot examine the originals or recreate quantitation reports/chromatograms. *The Audit* files in the electronic data showed 1 or 2 manual integration on the primary column and 2-5 manual integrations performed on the confirmation column. Manual integration might be used to reduce the % Difference (D) to achieve below 15% D limit. Without the *Epatemp* files, such action could not be documented. The first CCV met the <15% D limit, except surrogate Decachlorobiphenyl on the confirmation column (32%D). The second CCV showed 8 of the 17 peaks with %D>15% on the confirmation column. No CCV evaluation form was provided for the primary column. Since the CCV standards were not analyzed within the 12-hour window, the auditor did not calculate the % D for acceptability.
Method Blank	The "Sequence" file showed the first MB (11/19/00) was run with sample RZ781and the second MB (11/20/00) was run with sample RZ 776. No hard copy for both MBs and no electronic file for the second MB were available. The PCB results report indicated some positive aroclors responses but the final aroclors results were reported as NB. Not clear whether a MB was reported for each sample or only 1 MB was reported for both samples because MB and surrogate results were identical for both samples and the auditor had no raw data to check the quantitations. The extraction log indicated that the samples, MB, LCS/LCSD underwent florisil cleanup except samples RZ781 and RZ776. The MB should have been prepared exactly as the samples were prepared.
Laboratory Control Sample/Duplicate (LCS/LCSD)	The extraction log and the "Sequence file" indicated that the LCS/LCSD extracts were subjected to florisil cleanup while samples RZ781 and RZ776 were not. It was not clear to the auditor why a clean matrix of LCS/LCSD would use a florisil cleanup The % R for LCS/LCSD and the RPD were within the QC limits.
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	Hard copy/electronic data, summary data - not provided. Extraction log noted "not enough sample for MS/MSD."
Chain of Custody (COC), Sample Receipt, Standard, Extraction and Instrument Logs	No Standard Logs were provided
Client Sample	While 30 g of sample RZ776 was extracted, only 0.8 g of sample RZ781 was

QA/QC	SDG 0027 - Centrum - 8280 (PCBs) Analyzed 11/19 - 20/00 ID. Nos: RZ776, RZ781 2 - Soil
	extracted. No explanation was provided for the reduced weight. Detection limit was appropriately raised to reflect the reduced sample weight. All Aroclors were reported as Non -detect (ND). Surrogate tetrachloro-m-xylene (TCMX) recoveries met the QC limits. No recoveries were reported for surrogate decachlorobiphenyl (DCB). Although it was spiked and measured (manually integrated) in the client and QC samples, it appears that the recoveries were not reported because DCB failed the <15% D requirement on the second CCV. Due to the missing electronic files, failure to submit the ICAL data, failure to analyzed CCV standards at the required frequency, and missing or incomplete QC data, the auditor was not able to verify the acceptability of the aroclors results for samples RZ781 and RZ776.
Overview Comments	On the COC form for PCBs, the method was 8081and the laboratory used method 8080. The proper method should be 8082. This may be due to the method updates that occurred over time. No Method file was included in the electronic data, so the auditor could not review the instrument software specifications and quantitation approach.

TABLE 4: SDG RJ579 AUDIT SUMMARY

QA/QC	SDG RJ579 - Ceimic - 8270 (SVOC) Analyzed: 9/21/00, 9/26/00 ID. Nos: RJ592, RJ596, RJ599 and RJ587 reanalyzed 4 - Soil
Tuning	Ion abundance criteria used for 4 DFTPP masses were the ion abundance criteria specified in the Contract Laboratory Program (CLP) instead of the criteria specified in method 8270. The difference were as follows: Mass Method 8270 CLP 51 30-60% of mass 198 30- 80 of mass 198 127 40-60% of mass 198 25-75% of mass 198 365 >1.0% of mass 198 >0.75 of mass 198 443 17-23% of mass 442 15-24 of mass 442 Method 8270 allows the use of alternative tuning criteria provided the method performance was not adversely affected. Acceptable tunes were performed on the dates of ICAL (9/21/00) and CCV/client samples (9/26/00) analyses.
Initial Calibration (ICAL)	ICAL standards manual integrations were determined to be appropriate and accurately performed on the following: 10 ng Benzo(k)fluoranthene, benzo(g,h,i)perylene 20 ng Benzo(k)fluoranthene 80 ng Benzo(k)fluoranthene 120 ng Naphthalene, benzo(k)fluoranthene 160 ng Naphthalene, benzo(k)fluoranthene System Performance Check Compounds (SPCC) met minimum response factors (RF) limits and Calibration Check Compounds (CCCs) met the method requirement of <30% RSD for the RF of 5-6 levels of ICAL standards. RF of ICAL standards met 15% RSD limit except : % RSD Pyridine 17.2 Benzidine 107 Benzo(b)fluoranthene 18.6 Benzo(k)fluoranthene 21.0 Pyridine and benzidine were not present in the client samples but spiked and quantitated in the LCS. Benzo(b) and benzo(k)fluoranthene were present in the client sample RJ587. As the method specifies, for RSD >15%, average RF cannot be used for quantitation. Calibration curves should have been constructed for the 4 compounds rather than using the average RF. Consequently the results reported for these compounds should be considered estimates.
Continuing Calibration Verification (CCV)	CCV (9/26/00) met method requirements for CCCs and SPCCs
Method Blank MB	The following contamination were detected in two separate blanks: Affected samples Blank Contamination Concentration RJ587 Di-n-butylphthalate 48 ug/Kg Bis(2-ethylhexyl)phthalate 42 ug/kg RJ592, RJ596, RJ599 Di-n-butylphthalate 37 ug/Kg The phthalates (common laboratory contaminant) were present at very low concentrations.

QA/QC	SDG RJ579 - Ceimic - 8270 (SVOC) Analyzed: 9/21/00, 9/26/00 ID. Nos: RJ592, RJ596, RJ599 and RJ587 reanalyzed 4 - Soil
	MB applicable to RJ587 was spiked with surrogates at low concentrations appropriate for 8270 SIM. When reanalyzed using full scan GC/MS parameters (method 8270), surrogate levels were too low to be quantified.
Laboratory Control Sample/Duplicate (LCS/LCSD)	Recoveries of the LCS extracted 9/19/00 and analyzed 9/26/00 met the QC limits. No LCSD
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	Not provided . A MS/MSD were extracted and analyzed by 8270 SIM but results were not submitted with the 8270B.
COC, Sample Receipt, Standard, Extraction and Instrument Logs	Standard Logs - Not provided. COC, sample tracking, sample extraction and instrument logs provided were for 8270 SIM and not for 8270B. No other hard copy data or reporting forms were provided.
Client Sample	In the case narrative, sample RJ587 was initially extracted for 8270 SIM. However, the sample was reanalyzed for 8270B due to the high concentration of PAHs. By using method 8270B, no surrogates were identified/quantitated in RJ587 and the corresponding MB due to lower level spikes for the initial 8270 SIM. The organic data sheet showed for sample RJ587, di-n-butylphthalate and bis(2-ethylhexyl)phthalate were present in low levels (flagged "J"), but did not include a "B" flag indicating they are also present in the MB. Benzo(b)fluoranthene and benzo(k)fluoranthene were identified and quantitated. in sample RJ587. Since the RSD of these compounds exceeded the 15% limit and used average RF for quantitation and failed to construct a calibration curve for proper quantitation, the reported results should be considered estimates. In the case narrative, surrogate terphenyl-d14 was not recovered in sample RJ587 during 8270 SIM analysis and could be due to interference of high PAHs in the sample. Samples RJ592 and RJ596 contained no target compounds with the exceptions of low levels of phthalates. Sample RJ599 contained low levels of 2 phthalates and 350 ug/Kg of bis (2-ethylhexyl)phthalate. Surrogate % R were within acceptance criteria in all 3 samples. No hardcopy summary forms for tentatively identified compounds (TIC), although data for TIC were present in the electronic files.
Overview Comments	Overall, very few problems with the data.

TABLE 5: SDG RJ028 AUDIT SUMMARY

QA/QC	SDG RJ028 -Ceimic - 8270 SIM (PAHs) Analyzed 8/26/00, 8/28/00 ID. Nos: RJ28, RJ29, RJ33-RJ036, RJ038,RJ548, RJ550 and FJ552 9- Soil, 1-Water
Tuning	Ion abundance criteria used for 4 DFTPP masses were the ion abundance criteria specified in the Contract Laboratory Program (CLP) instead of the criteria specified in method 8270. The difference were as follows: Mass Method 8270 CLP 51 30-60% of mass 198 30- 80 of mass 198 127 40-60% of mass 198 25-75% of mass 198 365 >1.0% of mass 198 >0.75 of mass 198 443 17-23% of mass 442 15-24 of mass 442 Method 8270 allows the use of alternative tuning criteria provided the method performance was not adversely affected. Tunes were acceptable on ICAL (8/26/00), CCV and client samples (8/26/00 and 8/28/00).
Initial Calibration (ICAL)	The mean RFs and % RSD were reproduced by the auditor from the raw data for 2 surrogates and 12 of the 18 target compounds. All of the recalculated results match the ICAL standard data reported. The mean RFs for all compounds were >0.05. The CCCs met the % RSD limit of <30%. 12 target compounds and 1 surrogate exceeded the 15% RSD limit: % RSD % RSD N-Nitrosodimethylamine 17.2 Fluorence 18.3 Acenaphthylene 21.6 Pyrene 15.4 Anthracene 25 Benzo(b)fluoranthene 16.1 Benzo(a)anthracene 18.5 Indeno(1,2,3-cd)pyrene 19.6 Benzo(k)fluoranthene 19.4 Benzo(g,h,i)perylene 15.9 Dibenzo(a,h)anthracene 20.0 2-Fluorobiphenyl (surrogate) 20.0 2-Methylnaphthalene 18.1 Since the average RFs with > 15%RSD were used for quantitation , the results of the 12 target compounds were considered estimate. The laboratory should have constructed a calibration curve and use it for quantitation.
Continuing Calibration Verification (CCV)	The CCV standards (8/26 and 8/28/00) met method QC requirements.
Method Blank	The first MB and sample FJ552 (water) were extracted on 7/21/00. The second MB and the soil samples were extracted and underwent GPC cleanup on 7/26/00 Both MB met the QC limits.
Laboratory Control Sample/Duplicate	For water matrix, a LCS was analyzed and all 18 spiked compounds met the QC limits, although n-Nitrosodimethylamine was at the lower limit of

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(LCS/LCSD)	20% R. For soil matrix, the LCS recoveries of n-nitrosodimethylamine, anthracene, benzo(a)pyrene and benzo(g,h,i)perylene were below control limits.
Matrix Spike/Matrix Spike Duplicate	MS/MSD were not extracted/analyzed for either matrix Because no LCSD, duplicate sample or MS/MSD, there was no estimate of analytical precision documented by the laboratory for this SDG.
Chain of Custody (COC), Sample Receipt, Standard, Sample Extraction and Instrument Logs	COC, sample receipt and standard logs - Not provided The instrument run log was submitted and showed samples run on 8/26/00. It did not show tunes, standards and 8/28/00 samples.
Client Sample	Due to dilution, samples RJ036 (diluted 5X) and RJ036DL (diluted 10X) showed all 3 surrogate recoveries outside the laboratory recovery limits. The samples contained high levels of PAHs which caused extensive interference with surrogates. The failure to run the undiluted extracts or meet the % R criteria does not seriously impact data quality. Surrogate recoveries in all other QC and client samples met the QC limits. Results of target compounds with ICAL >15% RSD, except water sample FJ552 and soil sample RJ034 (both of the samples contained no PAHs above reporting limits) should be considered estimates. No hard copy summary forms provided for TICs, although data for TIC were present in the electronic files for each sample.
Overview Comments	Auditor's comments was based on Method 8270B requirements. Certain requirements in Method 8270B may or may not be applicable to the SIM data. The most important is ICAL requirement. If RSD of the ICAL is <15% RSD then average RF should be used for quantitation. If RSD exceeds 15%, a calibration curve should be constructed for quantitation. Six target compounds and 2 surrogates exceeded the 15% RSD limit and used the average RFs for quantitation. However, the project data and measurement quality objectives (DQO/MQO) might not require the 15% RSD limit for Method 8270 SIM analyses.
Overview Comments	The water matrix Recovery QC limits for 2 surrogates listed on the form did not comply with limits given in Method 8270B. Surrogate — Ceimic % R Limits — Method 8270B %R Limits 2-Fluorobiphenyl — 30-116 — 43-116 Terphenyl-d14 — 30-141 — 33-141 The method states that laboratories should established their own surrogate recovery limits but the limits must fall within the limits specified in Method 8270B. However, surrogate recoveries of the client and QC samples met the QC limits specified in the Method 8270B.

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	The upper limit of the ISTD areas listed on the summary ISTD forms is +150 of the area of ISTD in the daily (12-hour) CCV standard. In Methods 8270Band CLP Organics , the criterion for the upper ISTD area is + 100 of each CCV standard, so the basis for this deviation is unclear to the auditor. However, client and QC samples met the Method 8270B limit, +100 ISTD area limit.

TABLE 6: SDG 772 AUDIT SUMMARY

QA/QC	SDGRJ772 - Ceimic - 8270 SIM (PAHs) Analyzed 9/9/00, 11/13-15/00, 11/22/00, 11/27/00 RJ772 -RJ783, RJ788 -RJ791 16 soil
Tuning	Criteria used for the 4 DFTPP masses were the ion abundance criteria specified in the CLP instead of the criteria specified in method 8270. The differences were: Mass / Method 8270 / CLP 51 / 30-60% of mass 198 / 30-80 of mass 198 127 / 40-60% of mass 198 / 25-75% of mass 198 365 / >1.0% of mass 198 / >0.75 of mass 198 443 / 17-23% of mass 442 / 15-24 of mass 442 Method 8270 allows the use of alternative tuning criteria provided the method performance was not adversely affected. Tunes were acceptable for ICAL (9/9/00) , CCV/QC and sample/client sample analyses (11/13,11/14, 11/15, 11/22 and 11/27/00).
Initial Calibration (ICAL)	Mean RFs for all compounds were >0.05. The CCCs met the < 30% RSD limit. 6 target compounds and 2 surrogates exceeded the 15% RSD limit: %RSD Acenaphthylene 19.7 Benzo(k)fluoranthene 18.2 Dibenzo(a,h)anthracene 18.8 Anthracene 18.6 %RSD Indeno(1,2,3-cd)perylene 17.5 Benzo(g,h,i)perylene 15.4 2-Fluorobiphenyl (surrogate) 16.0 1,2-Dichlorobenzene (surrogate) 18.0
Continuing Calibration Verification (CCV)	All CCVs met QC limits.
Method Blank (MB)	3 MBs extracted and analyzed met the QC limits.
Laboratory Control Sample/Duplicate	One LCS (11/8/00) showed a recovery of 18% for n-nitrosodimethylamine,

QA/QC	SDGRJ772 - Ceimic - 8270 SIM (PAHs) Analyzed 9/9/00, 11/13-15/00, 11/22/00, 11/27/00 RJ772 -RJ783, RJ788 -RJ791 16 soil
(LCS/LCSD)	which was below the acceptance limits of 20% - 140%. Two other LCSs (11/15 and 11/16),showed recoveries of 30% and 45% for n-nitrosodimethylamine, which were within the acceptance limits. N-nitrosodimethylamine was not detected in the samples. However, use of those results should take into account the generally low recoveries of this analyte in the LCSs and the MS/MSD to be discussed. All other recoveries and surrogates met the QC limits.
Matrix spike/Matrix Spike Duplicate (MS/MSD)	Sample RJ774 was used for MS/MSD. In the 11/13/00analyses of RJ774 and MSD, the peak area of the internal standard perylene-d12 was slightly (0.10%) above the method-specified upper limit reported on Form VIII. Method 8270B requires corrective actions and sample reanalysis only if an ISTD area in a CCV standard falls outside the QC limits (-50% to +100%) set using the previous CCV standard. The area of the perylene-d12 peak has minimal impact on the data quality in this instance. 18 MS/MSD recoveries and RPDs met the QC limits, although the n-nitrosodimethylamine, dibenzo(a,h)anthracene and benzo(g,h,i)perylene were at the lower limit (20% R) in the MS.
Chain of Custody (COC), Sample Receipt, Standard, Sample Extraction and Instrument Logs	(COC), sample receipt and standard were not provided. Only 1 page of the MS instrument Log was submitted. This page listed the ICAL standards run from 9/11/00. No run log was submitted for the client or QC samples.
Client Samples	Six samples were analyzed at 5X or greater dilution. No reports or raw data submitted for undiluted extracts. Some observations are: RJ773(diluted 5X)- Surrogate results reported on the summary form met QC limits . However, all the surrogates were manually integrated (acceptably) and software flags on the quantitation report indicated that all 3 were below the limit of quantitation (BLOQ). Fluoranthene and chrysene were quantitated at 1 ug/Kg above the reporting limits. All other target compounds were reported as "U" This sample should have been analyzed undiluted. The sample ID on the raw data (chromatogram/quantitation report) was incorrectly given as RJ782. Other information of the raw data correlate properly with sample RJ773. RJ775 (diluted 5X) - Surrogate results reported met QC limits. However, 2 of the 3 surrogates were manually integrated (acceptably) and software flags the quantitation report indicated that those 2 surrogates were BLOQ. Target compounds were reported as "U". The sample extract should have been analyzed undiluted. RJ788(5X), RJ 789(5X) and RJ791(5X) - All 3 surrogate %Rs were outside the QC limits. The samples contained very high levels of PAHs. The extracts were also run at a 100X dilution. RJ790 (100X) - All 3 surrogate %Rs were outside the QC limits. The sample contained very high levels of PAHs . The extract was also run at a 1000X dilution. On the SV Organics Analysis Sheets, the flags used on the reported results (e.g. "J", "E", "D") were not explained. ISTD peak areas in samples RJ774, RJ788, RJ789 and RJ791 were not within -50% to +100% of the corresponding ISTD peak area in the CCV standard

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	for that 12-hour analytical sequence. Method 8270B requires a corrective action and reanalysis only if an ISTD area in a CCV standard falls outside those QC limits set using the previous CCV standard. All 5 CCV standards met the QC limits for the ISTD peak areas. The impact of the outlying ISTD in the samples are: RJ774 - ISTD perylene-d12 peak area was slightly above the metho specified upper limit reported on Form VIII (11/13/00). No PAHs, which would be quantitated using the perylene-d12, were identified i the sample. RJ788, RJ789, RJ791 - peak areas of ISTD phenanthrene-d10 were extremely high and the peak area of ISTD chrysense-d12 were very low. These measurements were adversely influenced by the presenc of high PAHs in the samples. Each samples was rerun as a dilution and all ISTD peak areas were acceptable, so there was no impact o the PAHs quantitation. Samples RJ775, RJ776 and RJ778-RJ781 contained no PAHs above the reporting limit. Samples RJ772-RJ774, RJ777, RJ782, RJ783 and RJ788-RJ791 contained PAHs. As mention in the ICAL, results of PAHs with ICAL >15% RSD limit should be considered estimates. TIC No hard copy summary forms provided for TICs, although data for TIC were present in the electronic files for each sample. No hard copy summary forms provided for TICs, although data for TIC were present in the electronic files for each sample.
Client Samples	No hard copy summary forms provided for TICs, although data for TIC were present in the electronic files for each sample.
Overview Comments	The auditor's comments were based on Method 8270B requirements . Certain requirements in Method 8270B may or may not be applicable to the Method 8270 SIM data. The most important is ICAL requirement. If RSD of the ICAL is <15% RSD then average RF should be used for quantitation. If RSD exceeds 15%, a calibration curve should be constructed for quantitation. Six target compounds and 2 surrogates exceeded 15% RSD limit and used the average RFs for quantitation. The project data and measurement quality objectives (DQO/MQO) might not require the 15% RSD limit for Method 8270 SIM analyses. Six samples were analyzed at 5X or greater dilutions. No report/raw data was submitted for undiluted samples. RJ773 contained low levels of PAHs and RJ775 contained no PAHs. These 2 samples should have been analyzed undiluted. The other 4 samples contained very high concentrations of PAHs and were diluted appropriately.

TABLE 7: SDG 214 AUDIT SUMMARY

QA/QC	SDG 214 - Ceimic - 8082 (PCBs) Analyzed 5/6-7/01, 5/10-11/01 (Dual columns) RJ214 - RJ216, RJ241, RJ243, RJ240(water) 5 -soil, 1-water
Initial Calibration (ICAL)	ICAL of aroclors 1016, 1242, 1254, 1260 and surrogates TCMX and DCB met the QC limit of <15% RSD.
Continuing Calibration Verification (CCV)	All CCVs were acceptable. The % difference between the average CF in the ICAL and CF in the CCV were within the acceptance limit of <15%. CCV standards fell within the established RT windows.
Method Blank (MB)	MBs for water and soil indicated no contamination.
Laboratory Control Sample/Duplicate (LCS/LCSD)	LCS met the QC limits for surrogate and aroclors spike recoveries.
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	A soil MS/MSD met the QC limits for surrogate recoveries, aroclor spike recoveries and RPDs.
Chain of Custody (COC), Sample Receipt, Standard, Sample Extraction and Instrument Logs	Standard log was not provided.
Client Samples	All samples met QC limits for surrogate recovery and RT. No manual integration performed. The software peak integration parameters were appropriate and resulted in consistent, accurate peak measurements.
Overview Comments	The case narrative incorrectly identified the columns used as DB-608 and DB-1701 when the forms and raw data listed the column as DB-5 and ZB-1701. The analytical work performed appears to be of very high

QA/QC	SDG 214 - Ceimic - 8082 (PCBs) Analyzed 5/6-7/01, 5/10-11/01 (Dual columns) RJ214 - RJ216, RJ241, RJ243, RJ240(water) 5 -soil, 1-water
	quality.